

Ruetgers-Nease

Chemical Company, Inc. • A subsidiary of Rütgerswerke AG



November 14, 1991

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Re: Centre County Kepone Site, Modifications to Mirex and Kepone
SOPs

As discussed during our November 13, 1991 telephone conversation, Ruetgers-Nease is requesting that the standard operating procedures (SOPs) for mirex and kepone be modified for the Centre County Kepone Site, State College, Pennsylvania. The modifications are based on laboratory (ENSECO-ERCO, Cambridge, Massachusetts) operational experience gained during the previous mirex and kepone analytical work. The modifications will allow the laboratory to run more efficiently, increasing mirex and kepone analytical capacity to 15 soil or 33 aqueous samples per week, while not affecting the data quality.

As another sample collection round is beginning today, Ruetgers-Nease is requesting an expedited review of these modifications (provided as an attachment to this letter). If you have any questions regarding the requested modifications, please feel free to contact us at (814) 238-2424.

Ralph E. Pearce

Ralph E. Pearce
Environmental Engineer

Sincerely,

Ian Bell

Ian Bell
Interim Project Coordinator

Attachment

cc: Bryan Smith, SMC Environmental Services Group, Inc.
Rock Vitale, Environmental Standards, Inc.
Dennis Flynn, ENSECO
Stevie Wilding, USEPA Region 3

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ORIGINAL
(Red)

Proposed Changes
for Analysis of
Mirex and Kepone
for Centre County Kepone Site,
State College, PA

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Page one of two pages

The following changes are to be amended to the SOPs for analysis of Mirex and Kepone in water, soil, tissue, and air samples.

Section 5. Change first sentence of introductory paragraph from "Mirex and kepone are extracted with methylene chloride from the aqueous matrix and analyzed by gas chromatography/electron capture negative ion mass spectrometry (GC/ECNIMS)." to "Mirex and kepone are extracted with methylene chloride from the aqueous matrix and analyzed by gas chromatography/electron capture negative ion mass spectrometry (GC/ECNIMS) after optional screening using gas chromatography with electron capture, flame ionization, or electron impact mass spectrometric detection." The screening procedures for all SOPs are included in Attachment A.

Section 8. Add to end of section "The 40 day analytical holding time is established for operational reasons and for consistency with other methods. Previous data indicates that the extracts are stable and the results are valid up to and beyond 100 days."

Section 9. Add to GC Screening "An electron impact GC/MS system may also be used."

Section 12.1. Change the first paragraph from:

"For perfluorotributylamine (PFTBA), the instrument calibration must be manually (hardware) verified and adjusted as necessary at the start of every 12 hours of operation. Ion abundance criteria are noted in the following chart. A mass spectrum of PFTBA is included in appendix A.", to:

"For perfluorotributylamine (PFTBA), the instrument calibration must be manually (hardware) verified and adjusted as necessary at the start of every 24 hours of operation. Ion abundance guidelines are noted in the following chart."

Section 12.3. Change "Acceptance criteria for the %D of the RRF for kepone relative to ¹³Cg-mirex are less than +40% for the ions used for quantitation." to "Acceptance criteria for the %D of the RRF for kepone relative to ¹³Cg-mirex are less than +40% compared to the average RRF from the initial calibration. If kepone is not detected, Values for the %D of Kepone less than -40% are acceptable because they indicate increased sensitivity for Kepone. If kepone is detected in a sample, the %D criteria for the associated continuing calibration is 40%."

Section 13.14. Change "If applicable, screen the extract by GC/ECD as outlined in appendix B." to "Screen the extract by GC/ECD, GC/FID, or GC/MS as outlined in Attachment A (of this document)."

AR303433

Page two of two pages

Section 14.1. Change GC temperature program from "40DEG C for 5.5 minutes, ramp up to 290DEG C at 10DEG/minute, and hold for 5 minutes. Under these conditions, kepone elutes close to two minutes before ¹³Cg-mirex; mirex and ¹³Cg-mirex coelute. ¹³Cg-mirex generally elutes between 23 and 27 minutes." to "200DEG C for 2.0 minutes, ramp up to 290DEG C at 10DEG/minute, and hold for 5 minutes."

Section 17. Under "Raw Data", add the following item. "-Screening chromatograms using GC/FID, GC/ECD, GC/EI/MS, GC/ECNIMS used to choose appropriate dilutions for analysis"

Accepted by:

Dave Byro, Remedial Project Director, U.S. EPA Region 3

Stevie Wilding, QA Manager, U.S. EPA Region 3

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